REPORT

Study Title:
Characterization of K32

Ricerca Study Number: 034706
Ricerca Document Number: 034706-1

Data Requirements:
OPPTS 830.6302 Color
OPPTS 830.6303 Physical State
OPPTS 830.6304 Odor
OPPTS 830.6316 Explodability
OPPTS 830.7000 pH
OPPTS 830.7050 / OECD 101 UV/visible light adsorption
OPPTS 830.7100 / OECD 114 Viscosity
OPPTS 830.7200 / OECD 102 Melting point/melting range
OPPTS 830.7300 / OECD 109 Density

Study Completion Date:
June 7, 2017

Author:
Lee Panter

STATEMENT OF NO DATA CONFIDENTIALITY CLAIM

No claim of confidentiality, on any basis whatsoever, is made for any information contained in this document. I acknowledge that information not designated as within the scope of FIFRA sec. 10(d)(1)(A), (B), or (C) and which pertains to a registered or previously registered pesticide is not entitled to confidential treatment and may be released to the public, subject to the provisions regarding disclosure to multinational entities under FIFRA sec. 10(g).

Company: Koch Agronomic Services

Company Agent: Eric A. Searcy

Signature: Eric A. Searcy

Date: 6/7/2017

GLP COMPLIANCE STATEMENT

The study reported herein, "Characterization of K32" Ricerca Study Number 034706, was conducted and reported in compliance with the Good Laboratory Practice Regulations set forth in Title 40, Part 160 of the Code of Federal Regulations of the United States of America.

L Pant — 6/7/17

Lee Panter, Study Director
Ricerca Biosciences, LLC

Date

Eric G Searcy — 6/7/2017

Eric Searcy, Sponsor Representative & Submitter
Koch Agronomic Services LLC

Date

QUALITY ASSURANCE UNIT STATEMENT

The Ricerca Quality Assurance Unit has performed inspections on the study, "Characterization of K32" Ricerca Study Number 034706. The results of these inspections, including any findings or observations, were reported to the Study Director and Management for appropriate corrective actions on the dates listed below:

Phase of Study Inspected	Dates Inspected	Dates Reported to Study Director	Dates Reported to Management
Protocol Review	Sep 12, 2016	Sep 12, 2016	Sep 12, 2016
Protocol Amendment	Nov 22, 2016	Nov 22, 2016	Nov 22, 2016
In-study Inspection	Oct 17, 2016	Oct 17, 2016	Oct 17, 2016
Data and Report Audit	May 17, 2017	May 17, 2017	May 17, 2017

Ann L. O'Leary

Ann L. O'Leary, Ph.D.
Quality Assurance Auditor

June 7, 2017
Date

APPROVALS

Study Title: Characterization of K32

Ricerca Document Number: 034706-1

Testing Facility: Ricerca Biosciences, LLC
7528 Auburn Road
Concord, OH 44077

Lee Panter, Study Director
Ricerca Biosciences, LLC

6/7/17
Date

Johnson Jutson, Ph.D., Management
Ricerca Biosciences, LLC

06/07/2017
Date

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ABSTRACT

In this study the physical and chemical properties of K32 were evaluated to meet the guidelines set forth by the United States Environmental Protection Agency's (EPA) Office of Prevention, Pesticides and Toxic Substances (OPPTS).

The physical-chemical properties of K32 evaluated in this study included: color; physical state; odor; explodability; pH; UV/visible light adsorption; viscosity; melting point/melting range; density [specific gravity]. These tests were conducted to meet the data requirements of the following guidelines: US EPA Product Properties Test Guidelines, OPPTS 830.6302, Color; OPPTS 830.6303, Physical State; OPPTS 830.6304, Odor; OPPTS 830.6316, Explodability; OPPTS 830.7000, pH; OPPTS 830.7050 ,UV/visible light adsorption; OPPTS 830.7100 Viscosity; OPPTS 830.7200 Melting point/melting range and OPPTS 830.7300; Density.

The results of the tests performed are summarized as follows:

- Color: Munsell Color Designation, 5PB9/1 (white)
- Physical State: Viscous Liquid
- Odor: Sulfurous
- Explodability: No explosions were observed for three trials at maximum height (32 ½ in).
- pH: 7.13
- UV/visible light adsorption:
  - pH 2: Maximum absorbance of 0.6813 (λ max) at 208.0 nm
  - pH 7: No maximum absorbance was detected at pH 7
  - pH 10: Maximum absorbance of 0.5059 (λ max) at 222.0 nm
- Viscosity: Analysis could not be performed due to the physical nature of the test substance.
- Melting point/melting range: Could not be determined as the test substance likely decomposes above 150 °C.
- Density (Specific Gravity): Analysis could not be performed due to the physical nature of the test substance.

STUDY INFORMATION

STUDY TITLE
Characterization of K32

RICERCA STUDY NUMBER
034706

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CONTRIBUTORS

These personnel participated in the study in the following capacities:

Lee Panter	Study Director - pH, Color, Odor, and Physical Characteristics
Joseph Emery	Associate Scientist I - Color, Odor, and Physical Characteristics
Melissa Grcar	Technician III - Color, Odor, and Physical Characteristics
Shayira Habeeb	Senior Scientist – UV/Vis
Julie Pelton	Associate Scientist - DSC

SCHEDULE OF EVENTS

Study Initiation:	Sept 23, 2016
Experimental Start Date:	Oct 5, 2016
Experimental Completion Date:	March 29, 2017
Study Completion Date:	June 7, 2017

RETENTION OF DATA

Upon completion of the study, the complete study file including all original raw data was submitted to the Ricerca Biosciences, LLC Archives for permanent storage following the provisions of GLP standards relevant to this study. Copies of representative raw data (as appropriate), shall be submitted to the Sponsor. All non-study specific raw data (e.g., instrument logs), a copy of the protocol, protocol amendments, and report shall be archived at Ricerca Biosciences, LLC.

INTRODUCTION

PURPOSE AND OBJECTIVES

The objective of this study is to characterize K32 for its color, physical state including appearance, odor, explodability, pH, UV/visible light absorption, melting point/melting range, viscosity and density.

REGULATORY COMPLIANCE

This study was conducted in accordance with the U.S. EPA Good Laboratory Practice (GLP) Standards, 40 CFR 160.

CONDUCT OF THE STUDY

The study was conducted at the Ricerca Biosciences, LLC AgChem Product Development Department Laboratories according to the Ricerca Biosciences, LLC protocol "Characterization of K32," document number 034706-0 and one amendment, located in Appendix A.

TEST SUBSTANCE

Test Substance Name:	K32-(Reaction products of NBPT with urea and formaldehyde)
Lot Number:	55700-30-13
NBPT Content:	17.3 wt%
Water Content:	2.4 wt% (Karl Fischer Analysis)

STORAGE AND DISTRIBUTION

The test substance was synthesized at Ricerca Biosciences LLC and was stored at refrigerated conditions. Upon receipt, the identity of the test substance was verified by comparison against included documentation. All preparations were made in a manner to preclude contamination or deterioration of the test substance. All preparations of the test substance were uniquely identified.

Upon completion of the study any portion of the test substance not utilized in the study will remain in storage at Ricerca Biosciences unless otherwise directed by the Sponsor.

ANALYTICAL PROCEDURES

Full details of each individual test method are contained below:

COLOR

Summary

The test was conducted to meet the data requirements of the US EPA Product Properties Test Guidelines, OCSPP 830.6302 Color.

Equipment

Munsell Book of Color, glossy finish, Production Run 88-A
Digital Temperature Indicator: Fisher# 14-649-84, s/n: 160131071

Procedure

The Munsell System is based on color-perception attributes of hue, lightness, and saturation. Munsell notation allows the color of opaque objects to be specified with respect to the following (in order): Munsell value-V, Munsell chroma-C, and Munsell hue-H. The color notation was obtained by visual comparison to Munsell value, chroma, and hue scales of the Munsell Book of Color. ASTM D 1535, "Method for Specifying Color by the Munsell System," was followed. Three people with normal color vision provided opinions. The analysis was performed at an average of 25.6 °C.

Results

The color of K32 was determined to be Munsell Color Designation 5PB 9/1 (white).

PHYSICAL STATE

Summary

The test was conducted to meet the data requirements of the US EPA Product Properties Test Guidelines, OCSPP 830.6303 Physical State.

Equipment

Digital Temperature Indicator: Fisher# 14-649-84, s/n: 160131071

Procedure

A brief description of the physical state of the test substance was based on a visual inspection. Conventional terms such as "solid," "granular," "mixture of liquid and solid,"

"liquid," "powder," "gas," and similar terms were used as appropriate to describe the substance. Since this is a subjective test, three people provided opinions. The analysis was performed at an average temperature of 25.4 °C.

Results
The physical state of K32 was determined to be a "viscous liquid."

ODOR

Summary
The test was conducted to meet the data requirements of the US EPA Product Properties Test Guidelines, OCSPP 830.6304 Odor.

Equipment
Digital Temperature Indicator: Fisher# 14-649-84, s/n: 160131071

Procedure
A brief description of the odor (or lack of it) was made. The odor was reported in descriptive terms such as "none," "slight," "characteristic of aromatic compounds," etc. Since this was a subjective test, three people provided opinions. The analysis was performed at an average temperature of 25.6 °C.

Results
The three observers judged the odor to be "sulfurous".

IMPACT EXPLODABILITY (SOLIDS)

Summary
Impact explodability was determined with the Bureau of Explosives Impact Apparatus as described in 49 CFR 173.53, Note 4. A small quantity of the test material was impacted with an 8-pound weight. The detail of the method will follow the guidelines of 44 Federal Register 16265 (March 16, 1979) and Bureau of Explosives Impact Apparatus, Method of Testing Solids, Association of American Railroads 17-1F (revised April 1979).

Equipment
Explosives Impact Apparatus

Procedure
- The test substance was allowed to reach room temperature.
- An amount of material was aliquoted into the copper cup and rotated to allow a uniform film to form on the base of the copper cup.
- The striker tip was inserted into the cup withdrawing to capture the cup on the striker.
- The striker cup was placed into the sample holder .
- The assembled sample holder was placed into the impact apparatus base.
- The 8 lb weight was dropped from a maximum height of 32 ½ in.

- Since no explosion was observed (flame/smoke/sound) the test was repeated for a total of three trials.

Results
No explosions were observed for three trials at maximum height (32 ½ in).

pH

Equipment
- pH Meter: Fisher AB150
- Environmental Chamber HQ-EC-00012, 25˚C, equipped with REMS sensor HQ-SENB-00827
- Sartorius Universal balance EMF0027
- 1 mL glass pipette

Solutions/Reagents
- D.I.U.F. Water: Fisher, Lot# 164351
- Buffer solution pH 4.00, Fisher
- Buffer solution pH 7.00, Fisher
- Buffer solution pH 10.00, Fisher

Procedure
- Approximately one liter of purified water was left to boil in a beaker on a hot plate for at least 15 minutes.
- The water was transferred to a 1-liter bottle capped and allowed to cool to room temperature.
- Once cool, 99 grams of the boiled, purified water was dispensed to each of three Erlenmeyer flasks.
- One gram of the test substance was transferred into each flask.
- The flasks were placed in a 25 °C chamber for one hour under constant agitation (stirring).
- pH was measured and the average of the three flasks was determined.

Results
The sample was tested for pH in triplicate (pH: 7.10, 7.15, 7.13); the average of the three replicates yielded a pH of 7.13.

ULTRAVIOLET-VISIBLE SPECTROPHOTOMETRY

Summary
The ultraviolet-visible (UV-vis) absorption spectrum is a quantitative measure of the ability of a substance to absorb radiation in the electromagnetic spectrum between approximately

200 and 800 nm. Absorbance in the UV-vis region temporarily promotes valence electrons to higher molecular orbitals. Generally, absorption in the UV-vis region is attributable to unsaturated groups, conjugated systems, or groups with non-bonding electrons.

Equipment

- GBC, double beam spectrophotometer
- Spectral Software, qualified
- Quartz photometric cells

Materials

- Acetonitrile: Fisher, Optima
- 1 N HCl: Fisher, Certified
- 1 N NaOH, Fisher, Certified
- Distilled Water

Stock Solution Preparation

- 118 mg of the test substance was weighed into a glass bottle and diluted with 100 mL of 50:50 (Acetonitrile:Water) yielding a solution of 1.18 mg/mL.

Sample Preparation

- 400 µL of the stock was aliquoted into a 20 mL scintillation vail + 9.6 mL of 1N HCl and vortexed to mix.
- 400 µL of the stock was aliquoted into a 20 mL scintillation vail + 9.6 mL of Distilled Water and vortexed to mix.
- 10.9 mg of test substance was weighed into a glass bottle and diluted with 200 mL of 1 N NaOH, yielding a concentration of 54.5 µg/mL

Procedure

- Solutions were prepared to target an absorbance of 0.5 to 1.5 units at the relevant lambda max.
- A calibrated UV-vis spectrophotometer was used to zero (background correct) the absorbance reading over the wavelength range 200–800 nm using quartz photometric cells.
- The UV-vis spectrum of the test substance solution was collected over the wavelength range 200–800 nm at ambient temperature in quartz photometric cells.
- Results are reported as maximum absorbance (λ_{max}).

Results

pH 2: Maximum absorbance of 0.6813 (λ_{max}) at 208.0 nm (Figure 1 & Figure 2).

pH 7: No maximum absorbance (λ_{max}) was detected for pH 7 (Figure 3 & Figure 4).

pH 10: Maximum absorbance of 0.5059 (λ_{max}) at 222.0 nm (Figure 5 & Figure 6).

VISCOSITY

Results

Due to the viscous nature of the test substance (very low flowability) the test substance is not amenable to running viscosity using the equipment available at Ricerca laboratories.

MELTING POINT/MELTING RANGE

Summary

Differential Scanning Calorimetry (DSC) measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes. The method is also considered unreliable if sublimation or decomposition occurs in the temperature range of interest. The current version of Ricerca Test Method TM-0092, "Determination of Melting Point and/or Purity by DSC", will be used as a guideline for this analysis.

Equipment

- Balance: Mettler XP205 DR
- TA Instruments DSC Model: Q2000
- Sample Pans & Lids: aluminum, crimped TA Tzero Hermetic Lid w/pin hole

Procedure

DSC Melting Point

- 4.17 and 5.37 mg of test substance was weighed into separate aluminum sample pans. The pans were sealed with an aluminum cover.
- Each sample pan was placed in the DSC sample holder. An empty sample pan was placed in the reference holder.
- Temperature region from 0 to 250 °C was scanned at 10 °C/min.
- The extrapolated onset value of the melting endotherm was recorded.

Results

A melting point/melting range could not be determined as DSC indicated decomposition of the test substance above 150 °C (see Figure 7).

SPECIFIC GRAVITY (RELATIVE DENSITY)

Results

Due to the viscous nature of the test substance (very low flowability) the test substance is not amenable to running specific gravity using the equipment available at Ricerca laboratories.

CONCLUSION

In this study the physical and chemical properties of K32 were evaluated to meet the US EPA Product Properties Test Guidelines, OPPTS 830.6302, Color; OPPTS 830.6303, Physical State; OPPTS 830.6304, Odor; OPPTS 830.6316, Explodability; OPPTS 830.7000, pH; OPPTS 830.7050,UV/visible light adsorption; OPPTS 830.7100 Viscosity; OPPTS 830.7200 Melting point/melting range and OPPTS 830.7300; Density.

Testing of K32 (lot number: 55700-30-13) generated results needed for the physical and chemical parameters described by the protocol for this study.

The results of the tests performed are summarized as follows:

- Color: Munsell Color Designation, 5PB9/1 (white)
- Physical State: Viscous Liquid
- Odor: Sulfurous
- Explodability: No explosions were observed for three trials at maximum height (32 ½ in).
- pH: 7.13
- UV/visible light adsorption:
  - pH 2: Maximum absorbance of 0.6813 (λ max) at 208.0 nm
  - pH 7: No maximum absorbance was detected at pH 7
  - pH 10: Maximum absorbance of 0.5059 (λ max) at 222.0 nm
- Viscosity: Analysis could not be performed due to the physical nature of the test substance.
- Melting point/melting range: Melting point/melting range: Could not be determined as the test substance likely decomposes above 150 °C.
- Density (Specific Gravity): Analysis could not be performed due to the physical nature of the test substance.

Figure 1: UV/Vis pH 2 Baseline

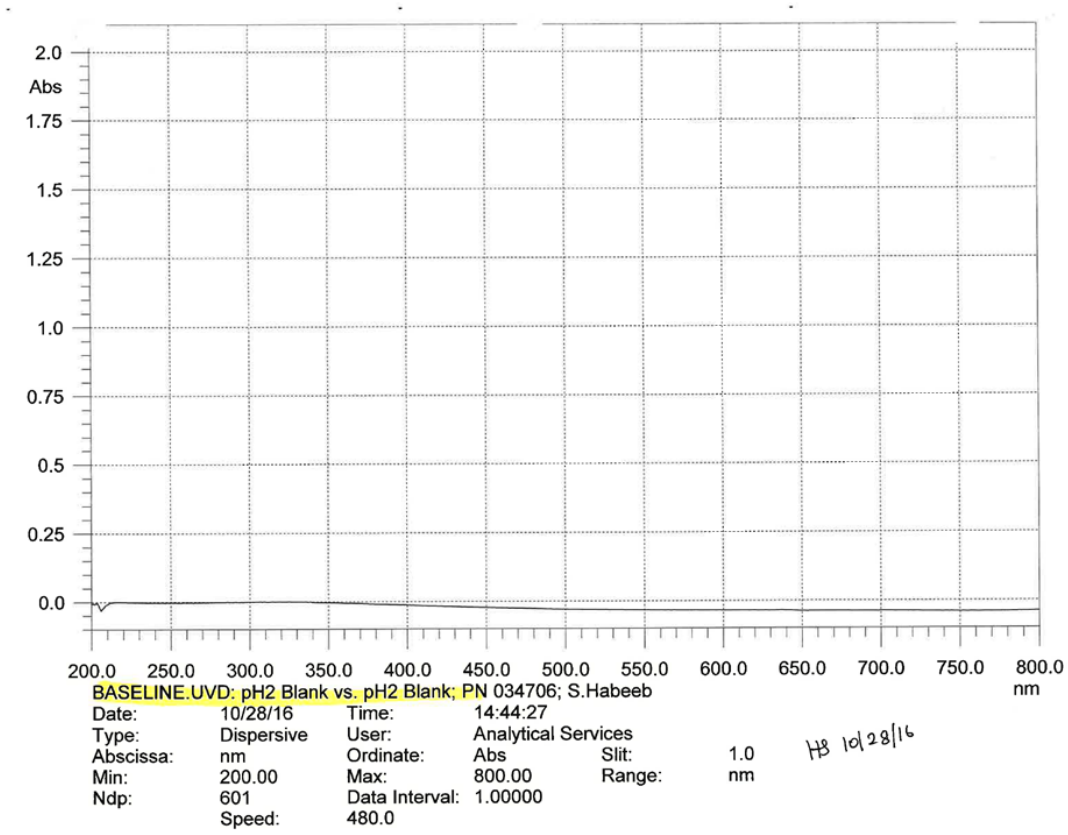


Figure 2: UV/Vis pH 2 Absorption

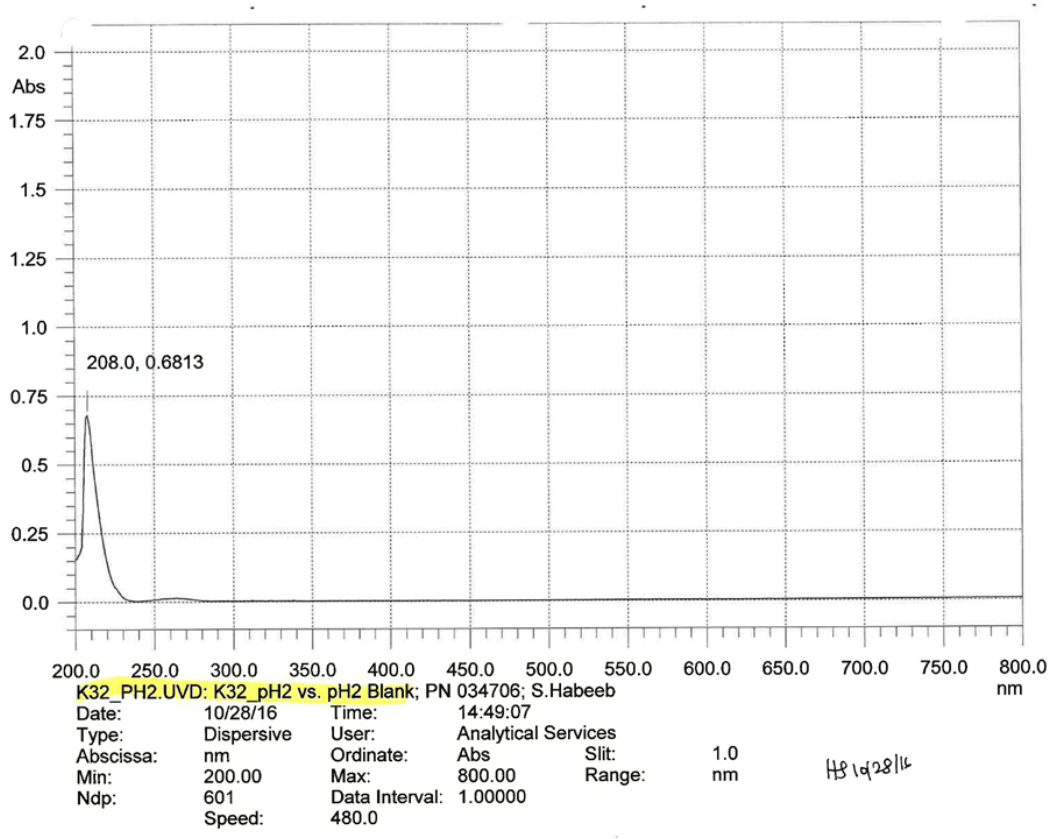


Figure 3: UV/Vis pH 7 Baseline

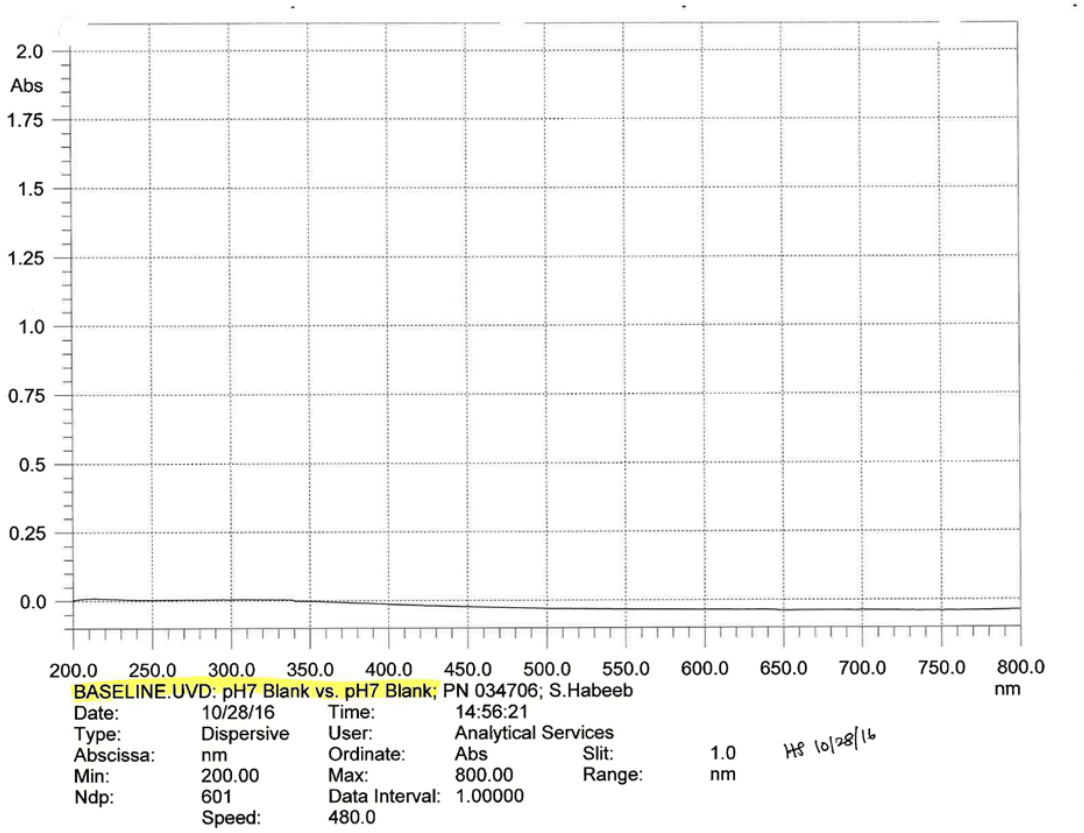


Figure 4: UV/Vis pH 7 Absorption

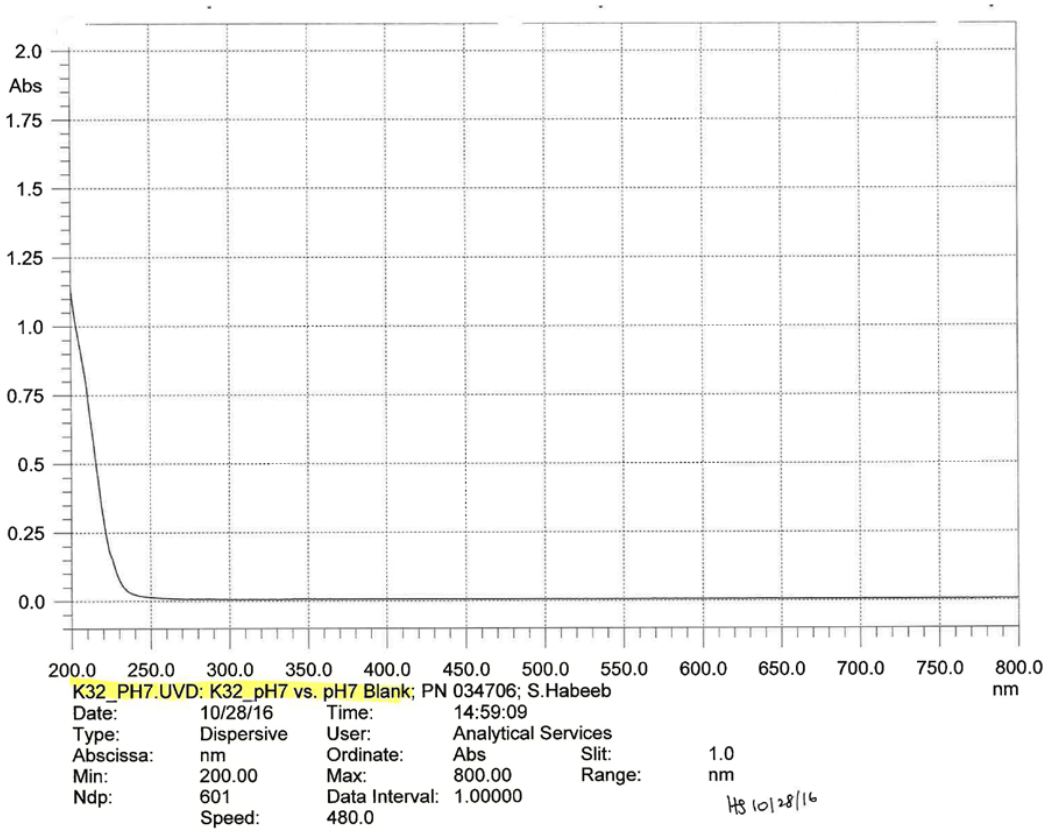


Figure 5: UV/Vis pH 10 Baseline

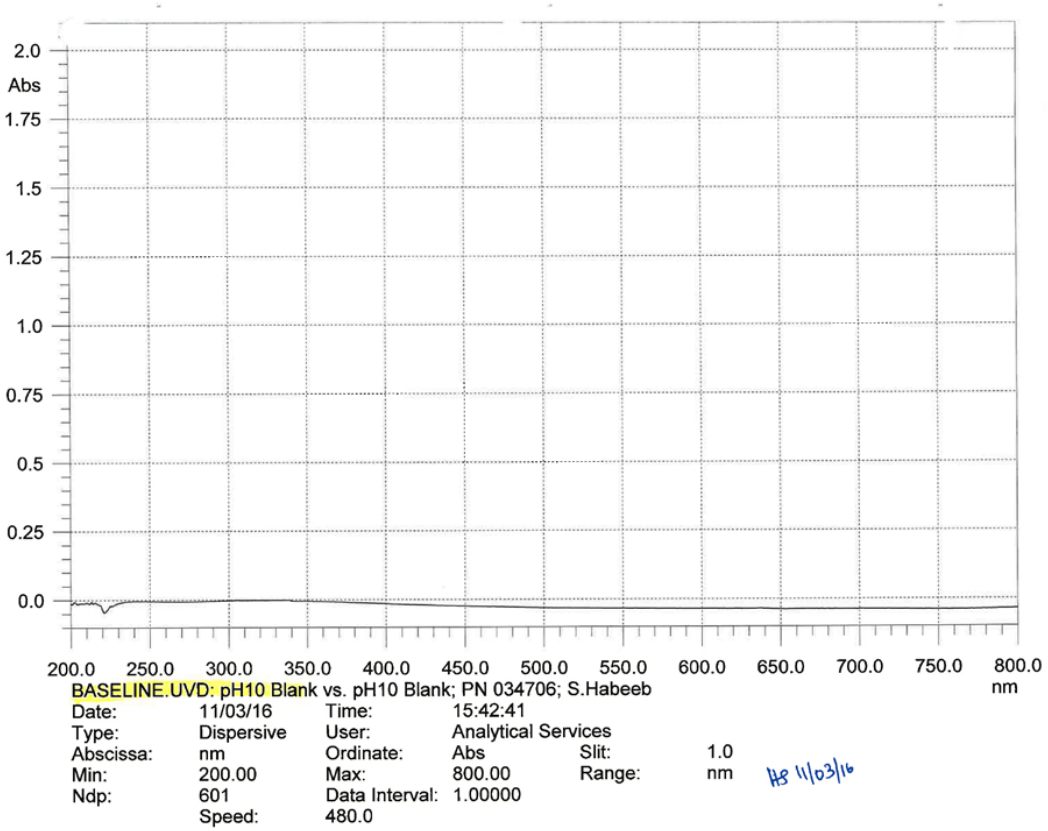


Figure 6: UV/Vis pH 10 absorption

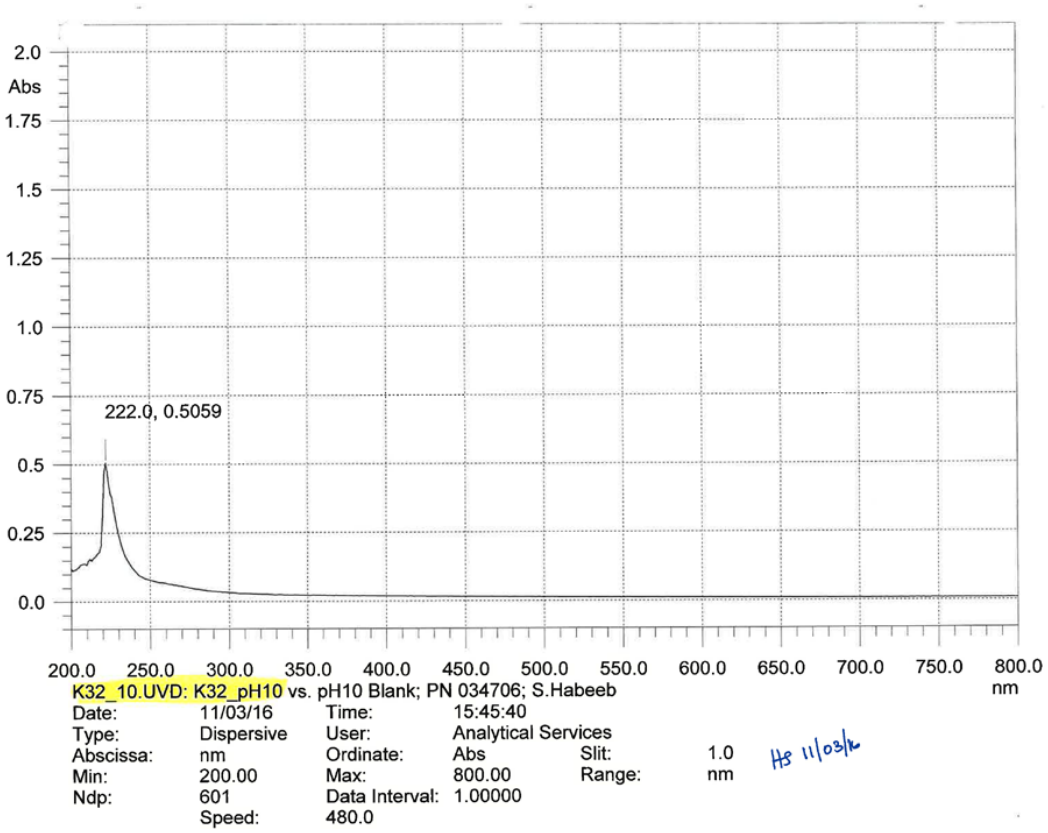
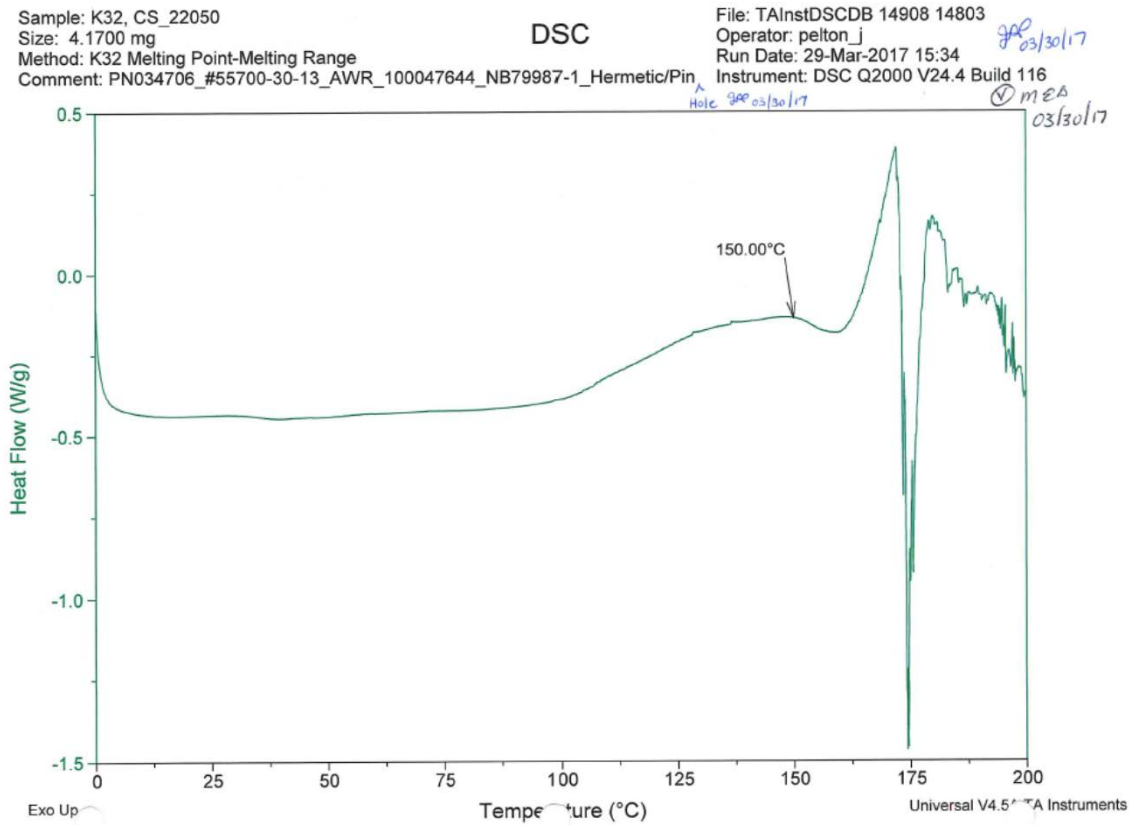


Figure 7: K32 – DSC



APPENDIX A

Protocol and Amendment

PROTOCOL

Study Title:

Characterization of K32

Ricerca Study Number: 034706
Ricerca Document Number: 034706-0

Data Requirement:

OPPTS 830.6302 Color
OPPTS 830.6303 Physical State
OPPTS 830.6304 Odor
OPPTS 830.6316 Explodability
OPPTS 830.7000 pH
OPPTS 830.7050 /OECD 101 UV/visible light adsorption
OPPTS 830.7100/ OECD 114 Viscosity
OPPTS 830.7200 / OECD 102 Melting point/melting range
OPPTS 830.7300 /OECD 109 Density

Testing Facility:
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Study Sponsor:
Koch Agronomic Services LLC
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STUDY INFORMATION

STUDY TITLE
Characterization of K32

RICERCA STUDY NUMBER
034706

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STUDY DIRECTOR
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PURPOSE AND OBJECTIVES
The objective of this study is to characterize K32 for its color, physical state; odor, explodability, pH, UV/visible light adsorption, viscosity, melting point/melting range, and density.

REGULATORY COMPLIANCE
This study will be conducted in accordance with the U.S. EPA Good Laboratory Practice (GLP) Standards, 40 CFR 160.

SCHEDULE OF EVENTS

Proposed Experimental Start Date:	September 2016
Proposed Experimental Termination Date:	October 2016

TEST SUBSTANCE

Test Substance Name:	K32-(Reaction products of NBPT with urea and formaldehyde)
Lot Number:	55700-30-13
NBPT Content:	17.3 wt%
Water Content:	2.4 wt% (Fischer Analysis)

STORAGE AND DISTRIBUTION

Ricerca will supply the test substance which will be stored at refrigerated conditions. Upon receipt, the identity of the test substance will be verified by comparison against included documentation. All preparations will be made in a manner to preclude contamination or deterioration of the test substance. All preparations of the test substance will be uniquely identified.

Upon completion of the study any portion of the test substance not utilized in the study will remain in storage at Ricerca Biosciences unless otherwise directed by the Sponsor.

ANALYTICAL PROCEDURES

Full details of each individual test method are contained below:

COLOR

Summary

The test will be conducted to meet the data requirements of the US EPA Product Properties Test Guidelines, OCSPP 830.6302 Color.

Equipment

Munsell Book of Color, glossy finish, Production Run 88-A
Traceable® Temperature Monitor, NIST traceable or equivalent

Procedure

The Munsell System is based on color-perception attributes of hue, lightness, and saturation. Munsell notation allows the color of opaque objects to be specified with respect to the following (in order): Munsell value-V, Munsell chroma-C, and Munsell hue-H. The color notation will be obtained by visual comparison to Munsell value, chroma, and hue scales of the Munsell Book of Color. ASTM D 1535, "Method for Specifying Color by the Munsell System," will be followed. Since this is a subjective test, three people with normal color vision will provide opinions. The analysis will be performed between 20 and 25 °C. The actual temperature will be reported.

PHYSICAL STATE

Summary

The test will be conducted to meet the data requirements of the US EPA Product Properties Test Guidelines, OCSPP 830.6303 Physical State.

Equipment

Traceable® Temperature Monitor, NIST traceable or equivalent

Procedure

A brief description of the physical state of the test substance will be based on a visual inspection. Conventional terms such as "solid," "granular," "mixture of liquid and solid," "liquid," "powder," "gas," and similar terms will be used as appropriate to describe the substance. Since this is a subjective test, three people will provide opinions. The analysis will be done between 20 and 25 °C. The actual temperature will be reported.

Odor

Summary

The test will be conducted to meet the data requirements of the US EPA Product Properties Test Guidelines, OCSPP 830.6304 Odor.

Equipment

Traceable® Remote Temperature Monitor, NIST traceable or equivalent

Procedure

A brief description of the odor (or lack of it) will be made. The odor will be reported in descriptive terms such as "none," "slight," "characteristic of aromatic compounds," etc. Since this is a subjective test, three people will provide opinions. The analysis will be performed at room temperature. The actual temperature will be recorded.

Impact Explodability (solids)

Impact explodability will be determined with the Bureau of Explosives Impact Apparatus as described in 49 CFR 173.53, Note 4. A small quantity of the test material will be impacted with an 8-pound weight. The detail of the method will follow the guidelines of 44 Federal Register 16265 (March 16, 1979) and Bureau of Explosives Impact Apparatus, Method of Testing Solids, Association of American Railroads 17-1F (revised April 1979). The experimentation will be conducted in at least duplicate.

Thermal Explodability

Equipment

- TA-DSC 2910 and controller
- Aluminum sample pans

Procedure

Thermal explodability will be determined per ASTM E-487 using a differential scanning calorimeter and open aluminum sample pans. Below is a summary of the method.

The dynamic screening test will be performed first. A 9 to 11-mg aliquot will be heated from 25 to 200 °C at a rate of 10 °C per minute in an air purge. The exotherm onset temperature will be noted. If there is no exotherm, the thermal explosivity test will be discontinued.

If there is an exotherm, the DSC will be cooled to room temperature, a new sample aliquot (9 to 11 mg) loaded, heated to 10 °C below the exotherm onset temperature, and held isothermally for 2 hours or until an exotherm occurs. If an exotherm occurs, test temperature will be decreased in 10 °C intervals (using a fresh sample aliquot each time) until no exothermic behavior is observed in the 2-hour period.

The test will be repeated with a 19 to 20-mg sample aliquot. It will be noted if a significant decrease in exotherm time is observed.

pH

Equipment

- Traceable® Remote Temperature Monitor, NIST traceable or equivalent
- Gel-filled polymer body combination electrode, Fisher #13-602-104 or equivalent
- Environmental chamber (25 °C)
- Magnetic stir plate
- Mettler PK300 balance (or equivalent) capable of measuring to the nearest 0.01 gram
- Assorted glassware

Reagents

- Purified water
- Buffer solution pH 4.00, Fisher certified SB98-500 or equivalent
- Buffer solution pH 7.00, Fisher certified SB108-500 or equivalent
- Buffer solution pH 10.00, Fisher certified SB116-500 or equivalent

Procedure

1. Boil approximately 1 liter of purified water for at least 15 minutes to remove dissolved gases. Allow to cool to room temperature in a tightly capped vessel to prevent the reabsorption of carbon dioxide.
2. Calibrate the pH meter
3. Measure 99 g of the boiled-out water into each of three 125-mL Erlenmeyers.
4. Add 1 g of the test substance to each of the three flasks and stopper.
5. While continually agitating, allow the three flasks to equilibrate at 25 °C for approximately 60 minutes. Record all applicable times and temperatures.
6. Remove the stopper from each flask and immediately measure and record the pH of each sample at 25 °C.
7. Check the calibration of the pH metering system by measuring and recording the pH of two buffer solutions. Be sure experimental values are within the range of calibration buffers.
8. Calculate an average from the three replicate samples.

Ultraviolet-Visible Spectrophotometry

Summary

The ultraviolet-visible (UV-vis) absorption spectrum is a quantitative measure of the ability of a substance to absorb radiation in the electromagnetic spectrum between approximately 200 and 800 nm. Absorbance in the UV-vis region temporarily promotes valence electrons to higher molecular orbitals. Generally, absorption in the UV-vis region is attributable to unsaturated groups, conjugated systems, or groups with non-bonding electrons.

Equipment

- UV-vis spectrophotometer
- Spectral Software, qualified
- Quartz photometric cells

Materials

- Solvent(s), ACS grade or suitable alternate

Procedure

1. Prepare a solution of the test article using a concentration that produces an absorbance of 0.5 to 1.5 units at the relevant lambda max.
2. Sonicate the solution of the test article to dissolve (if necessary).
3. Use a calibrated UV-vis spectrophotometer to zero (background correct) the absorbance reading over the wavelength range 200–800 nm using quartz photometric cells (or other appropriate wavelength range considering the solvent system used).
4. Collect the UV-vis spectrum of the test article solution over the wavelength range 200–800 nm at ambient temperature in quartz photometric cells (or other appropriate wavelength range considering the solvent system used).
5. It may be necessary to prepare additional solutions or dilute the test article solution to bring the solution into the desired absorbance range. Some compounds may not exhibit an absorbance maximum in the UV-vis wavelength range.
6. Calculate the molar absorptivity of any relevant absorption bands using the Beer-Lambert Law.

VISCOSITY

Apparatus

The method includes, but is not limited to, the following equipment:

- Brookfield LVT
- Brookfield RVT
- Brookfield LVDV-I Prime
- Brookfield LVDV-II + Pro
- Spindles
- Sample chambers/cups
- Brookfield Water Bath with temperature control, or equivalent
- Pipettes
- General laboratory glassware
- Thermometer
- Timer

Reagents, Supplies, Media, and Solutions
Brookfield Viscosity Standards or equivalent

Preparation of Calibration and Check Standards
Instrument is operated and calibrated as per the current version of SOP 19-C054.

Preparation of Samples
None

System Suitability

- Perform a standard check using a Brookfield viscosity standard at 25 ± 1 °C (as appropriate).
- The viscometer is suitable to use if the value obtained for the viscosity standard is within the acceptable range.
- Acceptable Range = ± (1% of the full scale range of the instrument +1% of the stated value of the viscosity standard)

  Example: *Calculate the acceptable range of viscosity using LVDV-II + Pro with CPE41 spindle at 60 RPM; Brookfield standard fluid 5 with a viscosity of 4.8 cP at 25 °C:*

  1) Calculate full scale viscosity range using the equation:

  *Full scale viscosity range [cP] = TK*SMC*10000/RPM*

  Where:

  TK = Viscometer torque constant = 0.09373 (from Operation manual)

  SMC = Current spindler multiplier constant = 1.228 (from Operation manual)

  RPM = Current viscometer spindle speed in RPM = 60

  *[cP] = 0.09373*1.228*10000/60 = 19.2 cP*

  The viscosity is accurate to (±) 0.19 cP (which is 1% of 19.2)

  2) The viscosity standard fluid is 4.8 cP. Its accuracy is (±) 1% of 4.8 or (±) 0.048 or 0.05 cP.

  3) Total allowable error is (0.05 + 0.19) cP = (±) 0.24 cP.

  Therefore, the acceptable range for 4.8 cP standard fluid is 4.8 ± 0.24 cP (4.56 – 5.04 cP) at 25 °C.

Procedure

1. Set up the water bath and connect to the viscometer sample chamber/cup. Adjust the water bath to hold the sample temperature constant at 25 ± 1 °C (as appropriate).
2. Attach the spindle to the viscometer if not already attached.
3. Transfer appropriate amount of sample into the sample cup and make sure no air bubbles are formed.
4. Attach the sample cup to the viscometer, allow the sample to equilibrate at the desired temperature, and record.
5. Select the spindle and set the speed as appropriate. Allow the spindle to rotate for at least five (5) times before viscosity reading is taken.
6. Record the viscosity value directly from the instrument display.
7. Repeat the analysis at 25 ± 1 °C.
8. Perform a viscosity calculation using the same procedure at a second temperature (to be determined by the analyst).

Calculations and Reporting

Record the viscosity of the fluid directly from the instrument display and report in cP.

Melting Profile By Differential Scanning Calorimetry

Summary

Differential Scanning Calorimetry (DSC) measures the amount of energy absorbed or released by a sample when it is heated or cooled, providing quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes. The method is also considered unreliable if sublimation or decomposition occurs in the temperature range of interest. The current version of Ricerca Test Method TM-0092, "Determination of Melting Point and/or Purity by DSC", will be used as a guideline for this analysis.

Equipment

- Differential scanning instrument
- Thermal Advantage Software, qualified
- Aluminum sample pan, covers, and crimper
- In-house N_2 or other specified purge gas
- Analytical balance

Procedures

DSC Melting Point

1. Weigh 2-10 mg of the test article into an aluminum sample pan. Seal the pan with an aluminum cover.
2. Place the sample pan in the DSC sample holder. Use an empty sample pan in the reference holder.
3. Scan the temperature region from 25 to 250 °C (or other appropriate range given the thermal behavior of the test article) at 10 °C/min.
4. Determine the extrapolated onset value of the melting endotherm and record as the melting point of the test article.

SPECIFIC GRAVITY (RELATIVE DENSITY)

Equipment

- 10-mL or other appropriate glass pycnometers with capillary top
- Analytical balance
- Water bath maintained at 25 °C
- NIST traceable temperature indicating device

Reagents

- Sterile Water for Inj., USP or equivalent carbon dioxide free water

Procedure

Reference CIPAC method MT 3

1. Weigh an empty and dry pycnometer with capillary top. Record the weight in grams as x.
2. Fill the pycnometer with the Sterile Water for Inj., USP and immerse to the top of the neck in the 25 °C water bath. Allow to equilibrate for at least 20 minutes.
3. Record the water bath temperature. Remove the pycnometer from the bath and immediately insert the stopper taking care not to introduce air bubbles, and gently push home with a slight twist.
4. Dry the pycnometer and top of the capillary taking care not to withdraw liquid from the capillary.
5. Reweigh the filled pycnometer and record the weight in grams as y.
6. Calculate the water equivalent (WE) or capacity of the pycnometer as follows:
   $WE = (y - x)$
7. Repeat the process with the test substance, taking care to avoid air bubbles when filling.

8. Calculate the Specific Gravity (relative density) as follows:

   Specific Gravity 25 °C/25 °C = (y' – x') / WE

   where:

   WE = water equivalent at 25 °C
   x' = weight of the empty pycnometer
   y' = weight of the pycnometer + test substance being examined

9. Analyze the sample in at least duplicate. Report the mean Specific Gravity (relative density).

PROPOSED STATISTICAL METHOD(S)

Appropriate statistical methods for the analysis and evaluation of the experimental data will be used at the discretion of the Study Director. Common statistical methods used to evaluate the precision and accuracy of the measurements may include (as appropriate): mean, coefficient of variation, standard deviation, and confidence interval.

To improve data presentation and interpretation, and facilitate report preparation, the Study Director may apply computer programs for spreadsheets (e.g., Excel), graphics presentations (e.g., Word or PowerPoint), and general standard statistics software.

RECORDS TO BE MAINTAINED

Analysts shall document all experimentation such that an experienced scientist can reconstruct the work. Documentation shall include sample identifications, weighings, dilutions, calculations, etc. Additional documentation shall include instrumentation and equipment utilized during the study, as well as documentation of prepared reagents and solutions.

All study data shall be reviewed or verified and maintained in folders in the study activity file. Research notebook(s) shall be placed in the study activity file at the completion of the study. Other comments, descriptions, calculations, correspondence, etc., shall be placed in the study activity file.

Upon conclusion of the study copies of representative raw data (as appropriate), shall be submitted to the Sponsor. An accurate study file, including original raw data, shall be submitted to the Ricerca Biosciences, LLC Archives, 7528 Auburn Road, Concord, Ohio.

REPORT

A final report will be prepared at the conclusion of the study. The report shall include, but not necessarily be limited to, the following:

- Name and address of the facility performing the study and the dates on which the study was initiated and completed, terminated, or discontinued
- Reference(s) to, and/or a detailed description of, all methods used
- Identification of the test and/or reference substances used in the study
- All deviations and changes from the protocol
- A description of all circumstances that may have affected the quality or integrity of the data
- Name and signature of the Study Director, the names of other scientists or professionals, and the names of supervisory personnel involved in the study
- Statistical methods employed for analyzing the data. A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data, and a statement of the conclusions drawn from the analysis.
- Locations where raw data and the final report are to be stored
- The signed and dated statement by the Ricerca Quality Assurance Unit specifying the dates of study inspections and dates the findings were reported to the Study Director and Management, when applicable.
- The signed and dated statement by the Study Director describing compliance with the Good Laboratory Practice Standards as specified in 40 CFR 160.

PROTOCOL DEVIATIONS

Deviations from the protocol, if any, will be documented and described in the raw data and reported.

PROTOCOL ACCEPTANCE

Study Title: Characterization of K32

Document Number: 034706-0

Testing Facility: Ricerca Biosciences, LLC
7528 Auburn Road
Concord, OH 44077

09/23/16
Lee Panter, Study Director — **Date**
Ricerca Biosciences, LLC

09/23/2016
Johnson Jutson, Ph.D, Management — **Date**
Ricerca Biosciences, LLC

9/23/2016
Eric Searcy, Sponsor Representative — **Date**
Koch Ag & Energy Solutions, LLC

PROTOCOL AMENDMENT ONE

Study Title:
Characterization of K32

Ricerca Study Number: 034706
Ricerca Document Number: 034706-0-1

Data Requirement:

OPPTS 830.6302 Color
OPPTS 830.6303 Physical State
OPPTS 830.6304 Odor
OPPTS 830.6316 Explodability
OPPTS 830.7000 pH
OPPTS 830.7050 /OECD 101 UV/visible light adsorption
OPPTS 830.7100/ OECD 114 Viscosity
OPPTS 830.7200 / OECD 102 Melting point/melting range
OPPTS 830.7300 /OECD 109 Density

Testing Facility:
AgChem Product Development
Ricerca Biosciences, LLC
7528 Auburn Road
Concord OH 44077

Study Sponsor:
Koch Agronomic Services LLC
2883 Miller Rd
Decatur GA 30035

DELETIONS ARE IDENTIFIED BY STRIKETHROUGH TEXT.
ADDITIONS ARE IN BOLD, UNDERLINED TEXT.

~~***THERMAL EXPLODABILITY***~~

~~**Equipment**~~

- ~~TA DSC 2910 and controller~~
- ~~Aluminum sample pans~~

~~**Procedure**~~

~~Thermal explodability will be determined per ASTM E-487 using a differential scanning calorimeter and open aluminum sample pans. Below is a summary of the method.~~

~~The dynamic screening test will be performed first. A 9 to 11 mg aliquot will be heated from 25 to 200 °C at a rate of 10 °C per minute in an air purge. The exotherm onset temperature will be noted. If there is no exotherm, the thermal explosivity test will be discontinued.~~

~~If there is an exotherm, the DSC will be cooled to room temperature, a new sample aliquot (9 to 11 mg) loaded, heated to 10 °C below the exotherm onset temperature, and held isothermally for 2 hours or until an exotherm occurs. If an exotherm occurs, test temperature will be decreased in 10 °C intervals (using a fresh sample aliquot each time) until no exothermic behavior is observed in the 2 hour period.~~

~~The test will be repeated with a 19 to 20 mg sample aliquot. It will be noted if a significant decrease in exotherm time is observed.~~

REASON FOR CHANGE

Melting Profile by Differential Scanning Calorimetry is already present in the protocol; as such thermal explodability is a redundancy and can be removed.

EFFECTIVE DATE

The date signed by the study director.

PROTOCOL AMENDMENT ACCEPTANCE

Study Title: Characterization of K32

Document Number: 034706-0-1

Testing Facility: Ricerca Biosciences, LLC
7528 Auburn Road
Concord, OH 44077

LPanter 11/23/16

Lee Panter, Study Director
Ricerca Biosciences, LLC

Date

11/23/2016

Johnson Jutson, Ph.D, Management
Ricerca Biosciences, LLC

Date

SPONSOR REPRESENTATIVE: ***ERIC SEARCY***

APPROVAL DATE: ***NOVEMBER 22, 2016***

SPONSOR: ***KOCH AGRONOMIC SERVICES LLC***